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IS/ISO 9917-2 (1998): Dental - Water-based cements, Part 2: Light-activated cements [MHD 8: Dentistry]

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भारतीय मानक

दंत्य — जल-आधारी सीमेंट

भाग 2 प्रकाश-सक्रियित सीमेंट

Indian Standard

DENTAL — WATER-BASED CEMENTS

PART 2 LIGHT-ACTIVATED CEMENTS

ICS 11.060.10

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

September 2008

Price Group 7

Dentistry Sectional Committee, MHR 08

NATIONAL FOREWORD

This Indian Standard (Part 2) which is identical with ISO 9917-2 : 1998 'Dental water-based cements — Part 2: Light-activated cements' issued by the International Organization for Standardization (ISO) was adopted by the Bureau of Indian Standards on the recommendation of the Dentistry Sectional Committee and approval of the Medical Equipment and Hospital Planning Division council.

Specific qualitative and quantitative requirements for freedom from biological hazard are not included in this part of standard, but it is recommended that when assessing possible biological or toxicological hazards reference be made to ISO 7405.

This standard is issued in two parts. Other part of the standard is:

Part 1 Powder/liquid acid-base cements

The text of ISO Standard has been approved as suitable for publication as an Indian Standard without deviations. Certain conventions are, however, not identical to those used in Indian Standards. Attention is particularly drawn to the following:

a) Wherever the words 'International Standard' appear referring to this standard, they should be read as 'Indian Standard'.

b) Comma (,) has been used as a decimal marker while in Indian Standards, the current practice is to use a point (.) as the decimal marker.

In this adopted standard, references appear to certain International Standards for which Indian Standards also exist. The corresponding Indian Standards which are to be substituted in their respective places are listed below along with their degree of equivalence for the editions indicated:

International Standard	*Corresponding Indian Standard*	*Degree of Equivalence*
ISO 3696 : 1987 Water for analytical use — Specification and test methods	IS 1070 : 1992 Reagent grade water — Specification (*third revision)*	Technically Equivalent
ISO 7491 : 1985 Dental materials — Determination of colour stability of dental polymeric materials	IS 12181 : 1987 Determination of colour stability of dental polymeric materials	do
ISO 9917 : 1991[1] Dental water-based cements	IS/ISO 9917-1 : 2003 Dental — Water-based cements: Part 1 Powder/liquid acid-base cements	do

The technical committee has reviewed the provisions of the following International Standards referred in this adopted standard and has decided that they are acceptable for use in conjunction with this standard:

International Standard	*Title*
ISO 3665 : 1996	Photography-Intra-oral dental radiographic film — Specification
ISO 7405 : 1997	Dentistry — Preclinical evaluation of biocompatibility of medical devices used in dentistry — Test methods for dental materials

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

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[1] Since revised in 2003 and adopted as IS/ISO 9917-1 : 2003.

Indian Standard

DENTAL — WATER-BASED CEMENTS

PART 2 LIGHT-ACTIVATED CEMENTS

1 Scope

This part of ISO 9917 specifies requirements for dental cements, including hand-mixed, encapsulated cements used for mechanical mixing and one-component materials, which are intended for base, lining and restoration purposes and for which the materials are water-based and set by multiple reactions which include an acid-base reaction and polymerization.

EXAMPLE Conventional glass polyalkenoate cements are normally formed by reacting an ion-leachable aluminosilicate glass with a polyalkenoic acid in an aqueous environment. Materials which fall within the scope of this part of ISO 9917 will normally be able to effect setting by such an aqueous acid-base type reaction, but in addition will be able to effect rapid setting through exposure to a suitable source of radiation.

NOTE — Attention of manufacturers and test houses is drawn to the closely related International Standards ISO 4049 and ISO 9917:1991. Consideration should be given as to the most appropriate International Standard by which to evaluate any individual product.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this part of ISO 9917. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this part of ISO 9917 are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 3665:1996, *Photography — Intra-oral dental radiographic film — Specification.*

ISO 3696:1987, *Water for analytical use — Specification and test methods.*

ISO 7491:1985, *Dental materials — Determination of colour stability of dental polymeric materials.*

ISO 9917:1991, *Dental water-based cements.*

3 Definitions

For the purposes of this part of ISO 9917, the definitions given in ISO 9917:1991 and the following definitions apply.

3.1 setting time
⟨in the absence of activating radiation⟩ period of time, measured from start of mixing, until the completion of set as defined in 7.3.1.

NOTE — In this part of ISO 9917, setting time is determined in the absence of activating radiation and is used to classify products as type I or type II.

3.2 initial hardening time
⟨in the absence of activating radiation⟩ period of time, measured from start of mixing, until a penetrator of given load and dimensions is unable to penetrate to within 0,1 mm of the bottom of a sample of cement 5 mm thick, as described in 7.3.2.

NOTE — In this part of ISO 9917 an estimate of working time is given by initial hardening time.

4 Classification

4.1 Clinical application

For the purposes of this part of ISO 9917, clinical application of these dental cements is classified as follows:

a) Bases and liners

b) Restorative cements

4.2 Setting reaction

For the purposes of this part of ISO 9917, these dental cements are classified on the basis of their setting characteristics as follows:

a) **Type I**: Products that undergo setting by light activation but also set in the absence of activating light (see 5.4 and 7.3.1).

b) **Type II**: Products that set only after application of activating light (see 7.3.1).

5 Requirements

5.1 Material

The cement may consist of a number of components that, when treated in accordance with the manufacturer's instructions, produces a material which will comply with the requirements of this part of ISO 9917.

When supplied as separate components for hand or mechanical mixing, the individual powder(s) and/or liquid(s) shall be free from extraneous matter (see 7.1.2). There shall be no visible signs of gelation in liquid components (see 7.1.2).

5.2 Sensitivity to ambient light

When tested in accordance with 7.2, there shall be no detectable change in the homogeneity of any of the three samples after being exposed to the test light for 30 s.

5.3 Setting time (in the absence of activating radiation)

When tested in accordance with 7.3, the setting time of type I materials in the absence of activating light shall be less than 60 min.

5.4 Initial hardening time [in the absence of activating radiation (Type I materials only)]

When tested in accordance with 7.3, the initial hardening time (measured in the absence of activating light) shall not be less than the value of the working time given by the manufacturer [see 8.3 f)].

5.5 Depth of cure

When tested in accordance with 7.4, the depth of cure shall not be less than 1 mm. If the manufacturer claims a greater depth of cure, the measured value shall not be more than 0,5 mm below the value stated by the manufacturer.

5.6 Flexural strength

When tested in accordance with 7.5, the flexural strength shall not be less than 20 MPa for restorative cements or 10 MPa for bases and liners.

5.7 Radiopacity (if claimed)

If the manufacturer claims that the material is radiopaque [see 8.2 d)], the radiopacity, determined in accordance with 7.6, shall be at least the same as the equivalent thickness of aluminium and not more than 0,5 mm below the value claimed by the manufacturer.

5.8 Opacity (restorative cements only)

When tested in accordance with 7.7, the cement shall have an opacity within the limits defined by the optical standards (i.e. between 0,35 and 0,90). This requirement does not apply to cements designated as opaque by the manufacturer [see 8.2 c)].

5.9 Shade and colour stability (restorative cements only)

When tested in accordance with 7.8, the shade of the material shall match closely that of the shade guide specified by the manufacturer.

When tested in accordance with 7.8 and ISO 7491, there shall be no more than a slight change in colour after 7 days.

6 Sampling

A sample drawn from one batch shall provide sufficient material to complete all the prescribed tests and any necessary repeats. The test sample shall consist of packages prepared for retail sale.

7 Test methods

7.1 Preparation of test specimens and test conditions

7.1.1 Test conditions

Unless specified otherwise by the manufacturer, prepare and test all specimens at a temperature of (23 ± 1) °C. Control the relative humidity to ensure that it remains greater than 30 % at all times. If the material was refrigerated for storage, allow it to attain (23 ± 1) °C.

Adequate provision shall be made for the exclusion of activating radiation when such conditions are required. Ambient light, both natural and artificial, is capable of activating these materials. For good control, the test should be performed in a dark room with any artificial light filtered by a yellow filter.

Water used in all tests specified in this part of ISO 9917 shall be prepared in accordance with ISO 3696 Grade 2.

7.1.2 Inspection requirements

Visual inspection shall be used in determining compliance with 5.1, 5.2, 5.8, 5.9 and clause 8.

7.1.3 Method of mixing

The cement shall be prepared according to the manufacturer's instructions. Sufficient cement shall be mixed to ensure that the preparation of each specimen is completed from one mix. A fresh mix shall be prepared for each specimen.

NOTE — For encapsulated materials, more than one capsule, simultaneously mixed, may be required for certain specimens. Likewise, for materials supplied in single-dose containers, several containers may be required for each test specimen.

7.2 Sensitivity to ambient light

7.2.1 Apparatus

7.2.1.1 Xenon lamp or radiation source of equivalent performance (a suitable apparatus is described in ISO 7491) with colour-conversion and ultraviolet filters inserted. The colour-conversion filter[1] shall be hardened glass 3 mm thick and shall have an internal transmittance which matches within ±10% that shown in figure 1.

The ultraviolet filter shall be made of borosilicate glass with a transmittance of less than 1 % below 300 nm and greater than 90 % above 370 nm.

NOTE — The purpose of the filter is to convert the spectrum of the xenon radiation, or equivalent, to that approximating a dental operating light. The filters and the output of the light should be checked periodically to ensure that the colour temperature at the luxmeter cell is 3600 K to 6500 K.

7.2.1.2 Two glass microscope slides/plates.

7.2.1.3 Illuminance-measuring device, e.g. luxmeter, capable of measuring illuminance of (8 000 ± 1 000) lx.

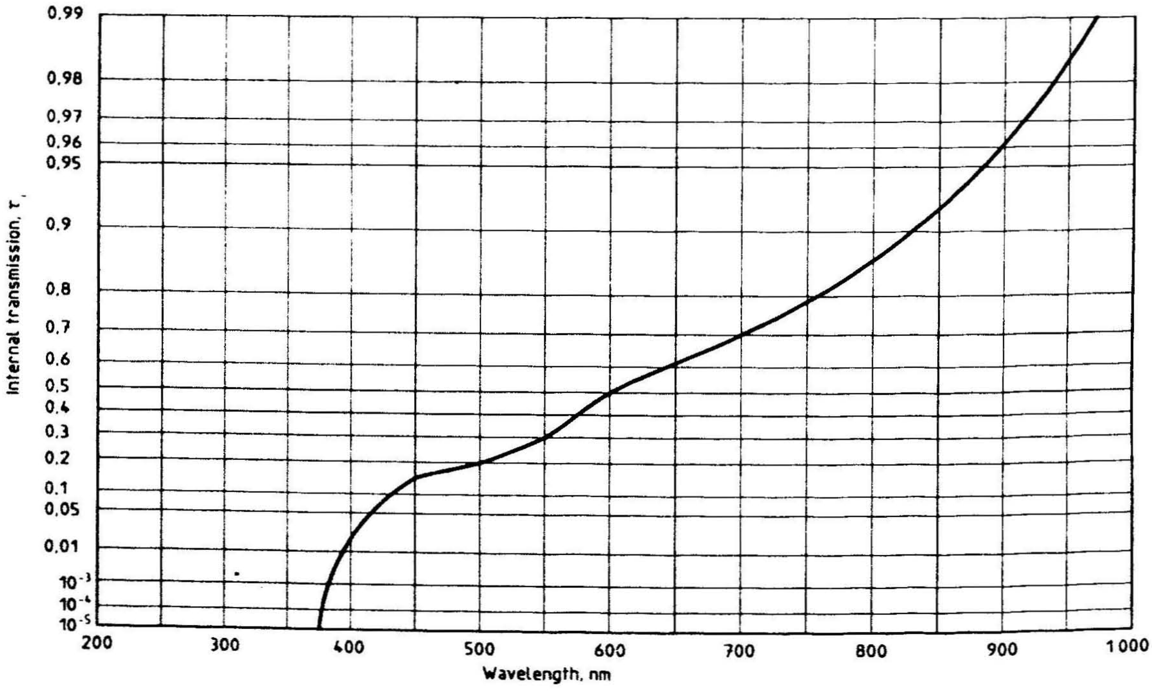

Figure 1 — Internal transmittance for colour conversion filter (see 7.2.1.1)

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[1] A suitable filter which corresponds to the internal transmittance shown in figure 1 and which is available commercially is the FG 15, hardened, rough-polished, 3 mm thick filter supplied by Schott Glaswerke, D-6500, Mainz 1, Germany. This information is given for the convenience of the users of this part of ISO 9917 and does not constitute an endorsement of this product by ISO.

7.2.1.4 Adjustable table.

7.2.1.5 **Matt black cover** for the luxmeter cell.

NOTE — This cover is intended to prevent reflection from the cell interfering with the observation of the experiment.

7.2.1.6 **Timer,** accurate to ± 1 s.

7.2.2 Procedure

In a dark room, position the illuminance-measuring device cell (7.2.1.3) under the xenon lamp with colour-conversion and ultraviolet filters inserted (7.2.1.1) at such a height as to provide an illuminance of 8 000 lx. The adjustable table (7.2.1.4) is needed to do this efficiently. Cover the cell with the black matt cover (7.2.1.5). Place 30 mg of material as a spheroidal mass on a glass slide (7.2.1.2), position the slide on top of the cell and expose it to the light for 30 s. Remove the slide with the sample from the irradiated area and immediately press the second microscope slide against the material with a slight shearing action to produce a thin layer.

Visually inspect the material to see whether it is physically homogeneous. The whole procedure shall be repeated twice using a new sample of material for each test. Record the results for all three tests.

NOTE — During this test, if the material has begun to set, clefts and voids will appear in the specimen when the thin layer is being produced. It may help to compare the tested material with a sample which has not been exposed to any activating radiation.

7.2.3 Treatment of results

If on visual inspection the material remains homogeneous for all three tests, the material is deemed to have satisfied the requirements of 5.2.

7.3 Setting time and initial hardening time

7.3.1 **Setting time,** in the absence of activating radiation (Type I materials only)

7.3.1.1 Apparatus

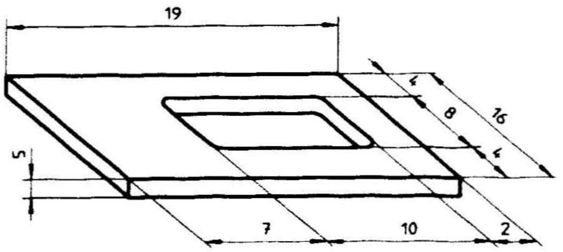
7.3.1.1.1 **Cabinet** capable of being maintained at a temperature of (37 ± 1) °C and a relative humidity of at least 90 %.

7.3.1.1.2 **Indentor,** of mass (400 ± 5) g and having a flat end of diameter (1 ± 0.1) mm. The needle tip shall be cylindrical for approximately 5 mm. The needle end shall be plane and perpendicular to the long axis of the needle.

7.3.1.1.3 **Metal mould** similar to that shown in figure 2.

7.3.1.1.4 **Metal block** of minimum dimensions 8 mm x 75 mm x 100 mm positioned within the cabinet and maintained at (37 ± 1) °C.

Dimensions in millimetres
Tolerance on dimensions: ± 0,15



NOTE — Internal corners may be rounded.

Figure 2 — Mould for preparation of specimens for determination of working time and setting time

7.3.1.1.5 Aluminium foil.

7.3.1.1.6 Timer, accurate to ± 1 s.

7.3.1.2 Procedure

NOTE — Material should be handled in the absence of light of wavelength 400 nm to 550 nm, e.g. using a dark room and/or filtered light.

Place the mould (7.3.1.1.3), conditioned to (23 ± 1) °C, on the aluminium foil (7.3.1.1.5) and fill to a level surface with mixed cement.

At 60 s after end of mixing, place the assembly, comprising mould, foil and cement specimen, on the block (7.3.1.1.4) in the cabinet (7.3.1.1.1). Ensure good contact between the mould, foil and block.

At 60 min after the end of mixing, carefully lower the indentor (7.3.1.1.2) vertically onto the surface of the cement. Allow it to remain there for 5 s and note whether the needle fails to perceptibly sink into the sample. Repeat the indentation at 60 min 30 s and note whether the needle fails to perceptibly sink into the sample and whether the second probing gives a result similar to the previous one (the definition of setting time in this test), when viewed using x2 magnification. Clean the needle between indentations. Note whether the material had set after 60 min.

Repeat the test twice. Note whether the material has set at 60 min for each experiment.

7.3.1.3 Treatment of results

Record the results of the three tests. For Type I materials, all three samples shall be set at 60 min (see 5.3).

7.3.2 Initial hardening time [in the absence of activating radiation (Type I materials only)]

7.3.2.1 Apparatus

As in 7.3.1.1 but without the environmental cabinet.

7.3.2.2 Procedure

As in 7.3.1.2 but the test is performed under the conditions described in 7.1.1.

Load the indentor 90 s after the end of mixing and then at 30 s intervals. Note the time from start of mixing at which the indentor fails to penetrate within 0,1 mm of the bottom surface of the cement. The initial hardening time is recorded as the time of the test indentation before this change is observed. Continue until the end of the working time [see 8.3 f)].

Repeat the test with two separate mixes of cement. Note whether the recorded initial hardening time is less than or greater than the working time stated by the manufacturer [see 8.3 f)].

7.3.2.3 Treatment of results

In order to satisfy the requirements (5.4), all three values of initial hardening time shall be at least as great as the value of working time stated by the manufacturer [see 8.3 f)].

7.4 Depth of cure

7.4.1 Apparatus

7.4.1.1 Stainless steel mould, for the preparation of a cylindrical specimen 6 mm long and 4 mm in diameter unless the manufacturer claims a depth of cure in excess of 3 mm; in this event, the mould shall have a depth of at least twice the claimed depth of cure.

NOTE — A mould release agent which does not interfere with the setting reaction, for example a 3 % solution of polyvinyl ether wax in hexane, may be used to facilitate removal of the set material from the mould.

7.4.1.2 Two slides/plates (glass or other suitable material), each of sufficient area to cover one end of the mould.

7.4.1.3 White filter paper.

7.4.1.4 Film, transparent to the activating radiation, (50 ± 30) µm thick, e.g. polyester.

7.4.1.5 External energy source, as recommended by the manufacturer for use with the test material [see 8.3 m)].

7.4.1.6 Micrometer, accurate to 0,01 mm.

7.4.1.7 Plastics spatula.

7.4.1.8 Timer, accurate to ±1 s.

7.4.2 Procedure

Place the mould (7.4.1.1) on a strip of the transparent film (7.4.1.4) covering the filter paper (7.4.1.3), and fill it with the test material prepared in accordance with the manufacturer's instructions. Take care to exclude air bubbles. Slightly overfill the mould and put a second strip of the transparent film on top. Press the mould and strips of film between the two glass slides (7.4.1.2) to extrude excess material. Remove the microscope slide covering one of the strips of film and gently place the exit window of the external energy source (7.4.1.5) against this strip of film. Irradiate the material for the time recommended by the manufacturer to achieve a depth of cure of at least 2 mm.

Remove the specimen from the mould immediately after completion of exposure and gently remove the uncured material with the plastics spatula (7.4.1.7). Measure the height of the cylinder of cured material with the micrometer (7.4.1.6) to an accuracy of ± 0,1 mm and divide this value by two.

Record this value as the depth of cure.

Repeat the test twice.

7.4.3 Treatment of results

If all three values are greater than 1 mm, the material satisfies the first requirement of 5.5.

In order to comply with the second requirement of 5.5, all three values shall be no more than 0,5 mm below the value stated by the manufacturer.

7.5 Flexural Strength

7.5.1 Apparatus

7.5.1.1 Mould, for the preparation of a test specimen (25 ± 2) mm x (2 ± 0,1) mm x (2 ± 0,1) mm. A suitable mould is illustrated in figure 3. The mould may be constructed from stainless steel or polytetrafluoroethylene. When stainless steel moulds are used, a release agent may be required to aid removal of specimens (see 7.4.1.1).

7.5.1.2 Two glass slides/plates (see 7.4.1.2), each of sufficient area to cover the mould.

7.5.1.3 Two stainless steel plates, each of sufficient area to cover the mould.

7.5.1.4 Small clamp.

7.5.1.5 Polyester film.

7.5.1.6 Water bath, capable of being maintained at (37 ± 1) °C.

7.5.1.7 External energy source(s), as recommended by the manufacturer for use with the test material [see 8.3 m)].

7.5.1.8 Micrometer, accurate to 0,01 mm.

Dimensions in millimetres

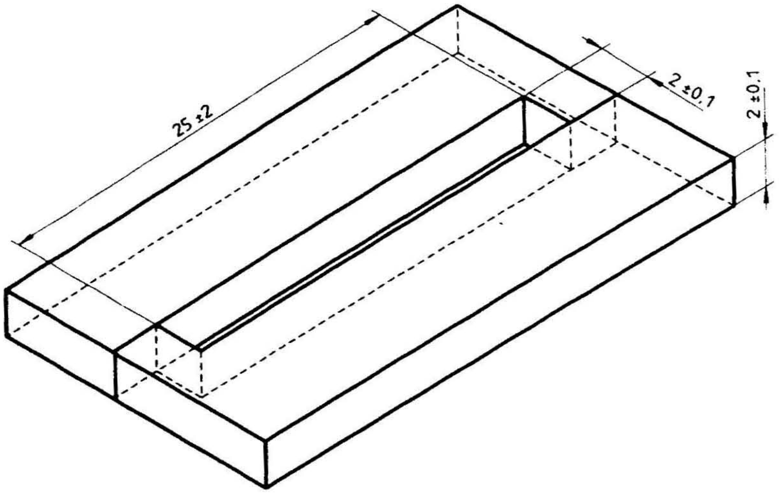


Figure 3 — Mould for preparation of specimens for determination of flexural strength

7.5.1.9 Flexural properties test equipment and apparatus, calibrated, to provide a constant crosshead speed of (0,75 ± 0,25) mm/min or a rate of loading of (50 ± 16) N/min.

The apparatus consists essentially of two rods (2 mm in diameter), mounted parallel with 20 mm between centres, and a third rod (2 mm in diameter) centred between, and parallel to, the other two, so that the three rods in combination can be used to give a three-point loading to the specimen.

7.5.2 Preparation of test specimens

Cover one of the metal plates (7.5.1.3) with polyester film (7.5.1.5) and position the mould (7.5.1.1) upon it. Prepare the material in accordance with the manufacturer's instructions and immediately place it in the mould. Place a second polyester film onto the material in the mould and cover this with a glass plate (7.5.1.2). Apply pressure to express excess material by means of the clamp (7.5.1.4). Place the exit window of the external energy source (7.5.1.7) at the centre of the specimen and against the glass plate. Irradiate that section of the specimen for the recommended exposure time. Move the exit window to the section next to the centre overlapping the previous section and irradiate for the appropriate time.

NOTE — The degree of overlap should incorporate not more than half of the previous area of irradiation covered by the light-activation probe.

Irradiate the section on the other side of the centre in the same way. Continue this procedure until the entire length of the specimen has been irradiated for the recommended exposure time. Repeat the irradiation procedure on the other side of the specimen (after switching the glass plate and metal plate). Place the assembly in the water bath maintained at (37 ± 1) °C, for 15 min. Then remove the specimen from the mould (mark the specimen at one end to indicate the face which was cured first) and store in distilled water at (37 ± 1) °C for (24 ± 1) h before testing.

Prepare five such specimens.

7.5.3 Procedure

Twenty-four (24 ± 1) hours after the start of irradiation, measure the dimensions of the specimen to an accuracy of ± 0,01 mm using the micrometer (7.5.1.8). Transfer the specimen to the flexural properties testing apparatus (7.5.1.9). The specimen shall be oriented such that the surface which was first exposed to the activating radiation is away from the applied force (i.e. in tension during the test).

At (24 ± 1) h after the start of irradiation and within 10 s of removing the specimen from the water bath (7.5.1.6), apply a load to the specimen at a crosshead speed of (0,75 ± 0,25) mm/min or at a rate of loading of (50 ± 16) N/min until the specimen fractures.

Record the maximum load exerted on the specimen.

Repeat the test on the four other specimens.

7.5.4 Calculation and expression of results

Calculate the flexural strength, σ, in megapascals, from the following equation:

$$\sigma = 3\frac{Fl}{2bh^2}$$

where

F is the maximum load, in newtons, exerted on the specimen;

l is the distance, in millimetres, between the supports, accurate to ± 0,01 mm;

b is the width, in millimetres, of the specimen measured immediately prior to testing.

h is the height, in millimetres, of the specimen measured immediately prior to testing.

7.5.5 Interpretation of results

Calculate the flexural strength results and report as follows.

a) If four of the results are above the limit specified in 5.6, the material is deemed to have complied with the requirements of 5.6.

b) If four of the results are below the limit specified in 5.6, the material is deemed to have failed.

c) If only three of the results are greater than the limit specified in 5.6, repeat the whole test. In the second series of tests all five specimens shall have a flexural strength equal to or greater than the value specified in 5.6, otherwise the material is deemed to have failed.

7.6 Radiopacity

7.6.1 Apparatus

7.6.1.1 Single-phase dental X-ray unit, with a total filtration of 1,5 mm aluminium, and capable of operation at (65 ± 5) kV with suitable accessories.

7.6.1.2 Dental X-ray occlusal film, of speed group D (in accordance with ISO 3665), with freshly prepared developing solution and fixer.

7.6.1.3 Aluminium step wedge, purity at least 98 % mass fraction with less than 0,1 % mass fraction copper and less than 1,0 % mass fraction iron present, 50 mm long x 20 mm wide, having a thickness range from 0,5 mm to 5 mm in equally spaced steps of (0,5 ± 0,01) mm. The wedge shall be free standing.

NOTE — The overall dimensions (50 mm x 20 mm) may be adjusted in relation to the film size, for the convenience of the user.

7.6.1.4 Photographic densitometer capable of measuring in the optical density range 0,5 to 2,5.

7.6.1.5 Mould for the preparation of specimen discs (15±1) mm in diameter and (1±0,1) mm thick.

7.6.1.6 Film, transparent to the activating radiation, (50±30) µm thick, e.g. polyester.

7.6.1.7 Glass slide/plates (see 7.4.1.2).

7.6.1.8 External energy source as recommended by the manufacturer for use with the test material [see 8.3 m)].

7.6.1.9 Micrometer, accurate to 0,01 mm.

7.6.1.10 Clamps.

7.6.2 Preparation of test specimens

Place the mould (7.6.1.5) on top of a glass plate (7.6.1.7) which is covered with film (7.6.1.6). Slightly overfill the mould with the material, prepared in accordance with the manufacturer's instructions. Place a piece of film (7.6.1.6) onto the material in the mould and cover this with the glass slide (7.6.1.7), thus expressing excess material. Briefly clamp (7.6.1.10) the assembly to ensure that a specimen of the correct thickness is produced. Remove the clamp and place the exit window of the external energy source (7.6.1.8) against the glass plate. Irradiate one section of the surface for the recommended exposure time. Repeat, using overlapping exposures, until the whole specimen has been irradiated for the recommended exposure time.

Remove the specimen from the mould and measure the thickness of the disc near its centre with the micrometer. Use only specimens whose thickness falls in the range (1,0 ± 0,1) mm.

7.6.3 Procedure

Position the X-ray film (7.6.1.2) on a sheet of lead not less than 2 mm thick. Place the specimen and the aluminum step wedge (7.6.1.3) in the centre of the film.

Irradiate the specimen, aluminium step wedge and film with X-rays at (65 ± 5) kV at a cathode-target film distance of 400 mm for such a time that, after processing, the region of film beside the specimen and aluminium has a photographic density of between 1,5 and 2.

NOTE 1 Exposures of between 0,3 s and 0,4 s at 10 mA are typical.

After developing and fixing the film, compare the density of the image of the specimen with that of the aluminium wedge using the densitometer (7.6.1.4).

NOTE 2 Improvement in the accuracy of this determination may be obtained by plotting a graph of optical density against thickness of aluminium for each exposure of the step wedge.

7.6.4 Interpretation of results.

NOTE — In interpreting the results of the radiopacity evaluation, it should be remembered that greater radiopacity produces a less dense (more transparent) image on the processed film.

If the density of the image of the specimen is less than the density of the image of the 1 mm aluminium step, the material is deemed to have complied with the first requirement of 5.7. In order to comply with the second part of 5.7, if a greater level of radiopacity is claimed, the equivalent thickness of aluminium shall be estimated from the film (take the nearest **higher** value of thickness from the aluminium wedge; e.g. if the specimen produces a film density equivalent to between 3,5 and 4,0 mm aluminium, the value of 4,0 is used). This value is compared with the value claimed by the manufacturer. In order to comply with the second part of 5.7, the value shall be no more than 0,5 mm less than the value claimed by the manufacturer.

7.7 Opacity (restorative cements only)

7.7.1 General

See 5.8 and 8.2 c) for the application of this test.

This test does not apply to cements which are designated as opaque by the manufacturer.

7.7.2 Apparatus

7.7.2.1 Opal glass standards with $C_{0,70}$ values of 0,35 and 0,90[2].

The contrast ratio $C_{0,70}$ is the ratio between the light reflected by the specimen on a black background and the light reflected by the specimen on a white background which has a reflectance of 70 %.

7.7.2.2 Sheets of white waterproof material of polyethylene or cellulose acetate (approximately 110 mm x 40 mm), marked along their entire length with black stripes 2 mm wide and 3 mm apart.

7.7.2.3 Mould as described in 7.6.1.5.

7.7.2.4 Clamps.

7.7.3 Preparation of test specimen

Prepare the specimens as described in 7.6.2.

After curing, carefully separate the specimen from the mould. Store the specimen for 7 d in water of Grade 2 according to ISO 3696, at (37 ± 1) °C.

7.7.4 Procedure

Compare the opacity of the specimen with the two appropriate opal glass standards (7.7.2.1) by placing the specimen and the standards on the black and white striped background. Cover the specimen, the opal glass standards and the sheets with a thin film of distilled water while making the comparison.

Alternatively, use a photometric instrument to make this comparison, provided that it can be demonstrated that the instrument has an opacity accuracy of within ± 0,02 $C_{0,70}$ In this procedure, place the specimen on an illuminated white background (reflectance 70 %) and measure the reflectance ($R_{0,70}$). Then transfer it to a black background and illuminate it with the same source and measure the reflectance (R_B). Calculate the opacity for the 1 mm thick disc from the equation:

$$C_{0,70} = \frac{R_B}{R_{0,70}}$$

7.7.5 Interpretation of results

Compare the opacity of the test disc with the two optical standards. To comply with 5.8, the opacity shall be in the range 0,35 to 0,90.

7.8 Shade and colour stability (restorative cements only)

7.8.1 Apparatus

7.8.1.1 Oven with water container or water bath capable of being maintained at (37 ± 1) °C.

7.8.1.2 Radiation source, water bath and other apparatus, as described in ISO 7491.

7.8.1.3 Film, (50 ± 30) µm thick, transparent to the activating radiation, e.g. polyester.

7.8.2 Preparation of test specimens

Prepare two disc specimens as described in 7.6.2.

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[2] Suitable opal glass standards may be obtained from the American Dental Association, 211 E. Chicago Ave, Chicago, IL, USA.

7.8.3 Procedure

Store one specimen in the dark, in water, for 7 d at (37 ± 1) °C; this is the reference specimen.

Store the second specimen in the same manner as the first (reference) for 24 h ± 2 h, then blank off half of the second specimen with aluminium or tin foil. Transfer this specimen to the radiation chamber, immerse it in water at (37 ± 5) °C and expose it to the radiation for 24 h. Ensure that the water level is (10 ± 5) mm above the specimen. After exposure, remove the metal foil, then transfer the specimen back to the oven (7.8.1.1) at (37 ± 1) °C and store it in the dark, in water, for 5 d. Compare the colour of both halves of the second specimen with each other and with the reference specimen and the manufacturer's nominated shade guide [see 8.2 b)]. Carry out the colour comparison in accordance with 5.9 and ISO 7491.

8 Packaging, marking and information to be supplied by manufacturer

8.1 Packaging

The components of the material shall be supplied in properly sealed containers which adequately protect their contents and have no adverse effect on the quality of the product.

An outer pack may also be used to present the containers as a single unit.

Information additional to that specified in 8.2 and 8.3 may be supplied at the discretion of the manufacturer or as required by legislation.

8.2 Marking

Each outer pack shall be clearly marked with the following details:

a) the name and/or trademark of the manufacturer, the type of cement (see clause 4) and/or whether the material requires light to activate setting and a statement (in language readily understood by the user) of the clinical application;

b) the shade of the material according to the manufacturer's nominated shade guide (for restorative cements);

c) if the material is designated 'opaque' (5.8), a clear statement to this effect;

d) if the material is designated 'radiopaque' (5.7), a clear statement to this effect;

e) the minimum net mass, in grams, of the powder and/or paste and the minimum net volume, in millilitres, of the liquid or paste, whichever is appropriate;

f) the manufacturer's batch or lot number;

g) if compliance with this part of ISO 9917 is claimed by the manufacturer, this may be indicated by marking the product with the number of this part of ISO 9917;

h) on the outermost packaging, the recommended conditions of storage, and the expiry date for the material under those conditions of storage.

In addition, each pack of encapsulated/pre-dosed cements shall be marked with the following information:

i) the number of capsules/pre-dosed containers in the pack, and the minimum net mass in each capsule/container.

NOTE — Information specified in 8.2 b) to 8.2 g) may be given on an inner leaflet or with the instruction leaflet, if this is more appropriate.

8.3 Manufacturer's instructions

Instructions shall accompany each package of the material and shall include the name and type (see clause 4) of the product and the following information, appropriate to the material (see 3.1):

a) precautions necessary to prevent inadvertent premature activation of setting;

b) the recommended powder/liquid ratio and instructions for use of any proportioning aids (e.g. scoops, etc.).

Also, for test purposes, the powder/liquid ratio on a mass/mass basis to a precision of 0,01 g at a temperature of (23 ± 1) °C and a relative humidity of (50 ± 10) % shall be included;

c) the condition and type of the mixing slab and spatula;

d) the rate of incorporation of the powder into the liquid;

e) the mixing time (see 3.1);

f) the working time;

g) if appropriate, a statement recommending that a liner be placed between the cement and the dentine;

h) the minimum time after irradiation at which finishing may be commenced;

i) the recommended method of finishing;

j) for encapsulated cements,the method of bringing about physical contact between the components;

k) the method, timing and type of mechanical mixing;

l) in the case of single-dose systems, the minimum net volume, in millilitres, of mixed cement in one container;

m) the recommended external energy source(s) and exposure times;

n) any special instruction relating to the proximity of the exit window of the energy source to the surface of the material;

o) the depth to which the material is cured after the recommended exposure (determined in accordance with 7.4).

Annex A
(informative)

Bibliography

[1] ISO 4049:1988, *Dentistry — Resin-based filling materials.*

[2] ISO 7405:1997, *Dentistry — Preclinical evaluation of biocompatibility of medical devices used in dentistry — Test methods for dental materials.*

[3] ISO 10993-1: –[3] , *Biological evaluation of medical devices — Part 1: Evaluation and testing.*

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[3] To be published. (Revision of ISO 10993-1:1992)

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